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TECHNICAL MEMORANDUM 52

EXPLOSIVE NATURE
OF DRY CALCIUM HYPOCHLORITE

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UNITED STATES ARMY
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FORT DETRICK

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TECHNICAL MEMORANDUM 52

EXPLOSIVE NATURE OF DRY CALCIUM HYPOCHLORITE

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FOREWORD

This publication is based upon exploratory research as a result of an accident in the U.S. Army Biological Laboratories. The findings are pertinent to other operations in the handling of etiologic agent.

ABSTRACT

The explosive nature of dry calcium hypochlorite has been established from a laboratory accident which occurred while autoclaving this compound with other laboratory materials. This report presents a series of investigations on the explosiveness of this compound. The reactions of this compound with water, oil, plastic, dibutyl phthalate, marking pen wick, and Class A combustible materials are discussed.

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I. INTRODUCTION

An explosion that occurred while steam sterilizing waste materials was reported to Industrial Health and Safety Division. The autoclave contained several pounds of dry calcium hypochlorite (CaOCl_2) in a plastic container, some oil, a felt marking pen, and a towel. Calcium hypochlorite, a strong oxidizing agent, was suspected of causing the explosion. Operating personnel had placed these materials in the autoclave to sterilize them before decontaminating the attendant cabinet systems.

Dry calcium hypochlorite, absorbent cotton, and vermiculite have long been used to pack and ship etiologic agents and biological materials. As much as 10 to 20 grams of dry calcium hypochlorite are used in each process. The dry calcium hypochlorite serves both as a decontaminant and as an absorbent in event of breakage or leakage.

In this instance the hypochlorite was left over from packaging etiologic agents for shipment. The other materials in the autoclave were used in previous operations. These were assumed to be waste materials, and were accordingly autoclaved before removing them from the biological cabinets. No cognizance was given to the thermal lability of hypochlorite powder or to the resultant interactions of the degradation products with other materials.

II. MATERIALS AND METHODS OF TESTING

Tests were conducted to investigate the explosive nature of dry calcium hypochlorite following the explosion. In these investigations, no attempts were made to simulate the conditions of the first explosion, nor was there any attempt to use more than five grams of dry calcium hypochlorite (Technical Type I, manufactured by Omega Chemical Corp.) in any test except that described in II, F. The materials to be tested were placed in a 100-milliliter beaker and autoclaved for 5, 15, or 30 minutes at 250°F and 20 pounds pressure. One thermocouple was inserted into the test materials, and another was placed in the autoclave to record the temperatures.

During the test, attempts were made to detect any explosion that might occur. The autoclave was pre-heated prior to each test to eliminate the noise caused by the contraction of the metals, which might be confused with the explosion produced by the materials. After the test all the materials were examined for signs of burning or decomposition.

A. TESTS OF DRY CALCIUM HYPOCHLORITE

One gram of dry 70 per cent calcium hypochlorite was placed in a 100-ml beaker and autoclaved at 250°F and 20 pounds pressure for five minutes. Neither burning nor explosion occurred during autoclaving although the calcium hypochlorite temperature increased to 270°F, 20°F higher than the autoclave temperature. However, after autoclaving the physical appearance of the calcium hypochlorite showed that it had decomposed.

B. TESTS OF DRY CALCIUM HYPOCHLORITE WITH OIL

One gram of dry 70 per cent calcium hypochlorite plus five drops of vacuum pump oil in a 100-ml beaker were autoclaved at 250°F and 20 pounds pressure for five minutes.

Explosions were audible when the autoclave temperature reached 245°F and 14 pounds pressure. Upon examination, the calcium hypochlorite in the beaker was found to be decomposed, and burning had taken place.

In similar tests, as little as one drop of oil was sufficient to cause a fire in the beaker; however, no explosions were detected in these tests.

C. TESTS OF DRY CALCIUM HYPOCHLORITE WITH PLASTIC MATERIALS

One gram of dry 70 per cent calcium hypochlorite plus small (one gram) pieces of plastic from the calcium hypochlorite container, were autoclaved in a 100-ml beaker at 250°F and 20 pounds pressure, for 5 and 15 minutes. Neither explosion nor burning occurred. The plastic materials remained unchanged, but the calcium hypochlorite was partly decomposed.

A similar test in which five grams of dry 70 per cent calcium hypochlorite plus five grams of plastic were autoclaved for 30 minutes at 250°F and 20 pounds pressure resulted in a heat gain in the calcium hypochlorite of 35°F over the autoclave temperature. The plastic deformed and the calcium hypochlorite decomposed.

In another test five grams of dry 70 per cent calcium hypochlorite plus five grams of plastic coated fiberboard (hatbox) material were autoclaved for 30 minutes at 250°F and 20 pounds pressure. These materials ignited after five minutes of autoclaving, resulting in the complete decomposition of the test materials.

A test similar to the first one using plastic, but including one drop of dibutyl phthalate (a plasticizing agent) in place of the plastic, resulted in explosions and burning of the materials. Explosions occurred when the autoclave reached a temperature of 145°F and a pressure of 13 pounds.

D. TESTS OF DRY CALCIUM HYPOCHLORITE WITH CLASS A COMBUSTIBLE MATERIALS

One gram of dry 70 per cent calcium hypochlorite and a small piece of towel (0.1 gram) were placed in a 100-ml beaker and autoclaved for 5 and 15 minutes at 250°F and 20 pounds pressure. Neither explosion nor burning occurred in either test. The hypochlorite was decomposed, and the temperature of the calcium hypochlorite was 35°F higher than that of the chamber.

In another test, five grams of dry 70 per cent calcium hypochlorite were mixed with some absorbent cotton (<0.1 gram) in a 100-ml beaker. This was autoclaved for 30 minutes at 250°F and 20 pounds pressure, and resulted in the burning of the cotton and complete decomposition of the calcium hypochlorite.

A test similar to that above but with the addition of 3 ml of water into the test materials did not produce any burning, but the calcium hypochlorite temperature increased 40°F higher than that of the autoclave. Again, the calcium hypochlorite completely decomposed; the cotton browned slightly.

When a similar test was carried out with the calcium hypochlorite in a cardboard shipping container instead of a beaker, burning took place inside the container with charring of the test materials. The lid of the container was blown off.

Another test involved autoclaving absorbent cotton alone. This did not cause a temperature increase in the cotton, nor was there any visible sign of degradation of the cotton after 30 minutes of autoclaving at 250°F and 20 pounds pressure.

E. TESTS OF DRY CALCIUM HYPOCHLORITE WITH MARKING PEN WICK

A small piece (<0.1 gram) of wick, clipped from a marking pen, and one gram of dry 70 per cent calcium hypochlorite were autoclaved for five minutes at 250°F and 20 pounds pressure. The test materials exploded and burned in less than three minutes of autoclaving.

F. TESTS OF DRY CALCIUM HYPOCHLORITE WITH WATER

Ten grams of dry 70 per cent calcium hypochlorite were placed in a cardboard shipping container with some absorbent cotton (0.1 gram). Ten milliliters of water were introduced into this container. The temperature inside the container increased 30°F indicating an exothermic reaction had occurred.

III. RESULTS

Dry calcium hypochlorite, when mixed with various materials and steam sterilized, will undergo thermal decomposition at temperatures of about 250°F, presumably with the liberation of the oxygen and chlorine that make it a strong oxidizing agent. The reaction is exothermic. Because this material is a strong oxidizer, it will react violently with such substances as oil, marking pen wick, and dibutyl phthalate (Table I).

TABLE I. REACTION OF ONE GRAM OF DRY CALCIUM
HYPOCHLORITE (70%) WITH VARIOUS TYPES OF MATERIALS

1 gm CaOCl plus	Autoclave Time, min. ^{a/}	Results			
		Explosion	Burning	Heat Gain ^{b/}	Decomposition of CaOCl
Oil (1 drop)	5	-	+	+	+
Oil (5 drops)	5	+	+	+	+
Dibutyl phthalate (1 drop)	5	+	+	+	+
Marking pen wick (<0.1 gm)	5	+	+	+	+
Plastic (1 gm)	5	-	-	+	+
	15	-	-	+	+
Towel (0.1 gm)	5	-	-	+	+
	15	-	-	+	+
CaOCl alone	5	-	-	+	+

a. Time after reaching 250°F and 20 pounds pressure.

b. Heat gain indicates temperatures above 250°F.

With regard to Class A combustible materials, such as towel and cotton, neither burning nor explosion was noticed when the dry calcium hypochlorite and these materials were autoclaved for 5 or 15 minutes at 250°F. However, when these same materials were autoclaved for 30 minutes under the same conditions, combustion resulted (Tables I and II).

TABLE II. REACTION OF FIVE GRAMS OF DRY CALCIUM HYPOCHLORITE (70%) WITH VARIOUS TYPES OF MATERIALS

5 gm CaOCl ₂ plus	Autoclave Time, min. ^a /	Results			
		Explosion	Burning	Heat Gain ^b /	Decomposition of CaOCl ₂
Plastic (5 gm)	30	-	-	+	+
Fiberboard (hatbox, 5 gm)	30	-	+	+	+
Absorbent cotton (<0.1 gm)	30	-	+	+	+
Absorbent cotton (<0.1 gm) plus water (3 ml)	30	-	-	+	+
Absorbent cotton ^c / (<0.1 gm) plus water (3 ml)	30	-	+	+	+

a. Time after reaching 250°F and 20 pounds pressure.

b. Heat gain indicates temperatures above 250°F.

c. This test was done in a closed cardboard shipping container instead of a beaker.

IV. DISCUSSION

Dry calcium hypochlorite is a highly unstable compound. When this compound is heated to 250°F and 20 pounds pressure, it invariably undergoes thermal decomposition accompanied with elevation of temperature. In the presence of such organic materials as oil, dibutyl phthalate (plasticizer) or felt marking pen ink, explosion and burning can be detected. Other violent reactions result when hypochlorite reacts with cellulosic materials at 250°F. Reactions of this type can be reduced so that no burning occurs if these materials are wetted with water. However, burning can occur if these materials react in a closed cardboard container. Burning also occurs when calcium hypochlorite reacts with plastic-coated fiberboard.

No significant change occurs in the reactivity of plastic material with dry calcium hypochlorite at 250°F, but an elevation of 35°F in temperature can be noted.

Similarly, the reaction of dry calcium hypochlorite with water in a closed cardboard container at room temperature produces a heat gain of 37°F.

V. CONCLUSIONS

The explosive nature of dry calcium hypochlorite, together with the supporting evidence reported in these tests, shows that hypochlorite should not be autoclaved in the presence of organic material. These precautions are especially necessary if etiologic agents packaged in hypochlorite and organic materials must be heated or autoclaved before disposal.

REFERENCE

Sax, N.I. "Calcium chloride hypochlorite," in "Dangerous properties of industrial materials," Reinhold Publishing Corporation, New York, 1957. pp. 425-426.

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